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STRUCTURE AND MECHANICAL PROPERTIES OF NANOSTRUCTURED Al-0.3%Cu ALLOY

Aneela Wakeel*, Tianlin Huang *, Guilin Wu*, Tianbo Yu**,
Niels Hansen** and Xiaoxu Huang **

*College of Materials Science and Engineering, Chongqing University,
Chongqing 400045, China

**Danish-Chinese Center for Nanometals, Section for Materials Science and Advanced
Characterization, Department of Wind Energy, Technical University of Denmark, Risø Campus,
DK-4000 Roskilde, Denmark.

ABSTRACT

An Al-0.3%Cu alloy has been produced using extremely high purity (99.9996%) Al and OFHC Cu. The alloy was cold rolled to 98% thickness reduction, forming a stable lamellar structure that has a lamellar boundary spacing of about 200nm and a tensile strength of 225MPa. During recovery annealing at temperatures up to 150°C, slight structural coarsening occurred without changing the lamellar structural morphology. At the same time the strength decreased, but no obvious enhanced tensile instability was observed as reported in the annealed nanostructured commercial purity Al1050 and Al1100. A good combination of yield strength as high as 100 MPa and elongation of larger than 10% could be obtained after annealing at medium temperatures where partial recrystallization took place.

1. INTRODUCTION

Nanostructured or ultrafine grained commercial purity Al alloys has been successfully produced by high strain deformation and as a result the strength has been significantly enhanced as compared with their coarse grained counterpart (Tsuji, Ito, Saito and Mianamino 2002; Hoppel, May and Goken 2004; Yu, Kao and Chang 2005; Kim, Kang and Shin 2006). For example, two commercial purity alloys, Al1050 (99.5% pure) and Al1100 (99.0% pure) have been processed by accumulative roll bonding (ARB) to a strain of about 5 (Kamikawa 2005; Huang, Hansen and Tsuji 2006), increasing the ultimate tensile strength (UTS) from less than 100MPa to more than 150MPa. A nanoscale lamellar structure has been developed in both alloys, however, it has been found that the degree of structural refinement and the strength reached are rather different for the two alloys: (i) The lamellar boundary spacing in the as-ARB processed state was reported to

be about 400 nm for Al1050 (Kidmose, Lu, Winther, Hansen and Huang 2012) and be about 200 nm for Al1100 (Kamikawa 2005; Huang et al. 2006) and (ii) The UTS is about 160 MPa for Al1050 (Kidmose et al. 2012) and about 300-330 MPa for Al110 (Kamikawa 2005; Huang et al. 2006). This significant difference in structural refinement and strength enhancement is not yet fully understood. One reason may be a slight difference in the content of substitutional solute Cu, which is <0.05wt% in Al1050 and is in the range of 0.10 to 0.15wt% in Al1100. Such a difference has a negligible effect on the strength in the soft condition indicating that the solid solution hardening by the small amount of Cu is insignificant. However, this difference may play a role in the evolution of the microstructure during plastic deformation and as a consequence affect the mechanical properties.

In this study we design an Al-0.3wt% Cu alloy, to investigate the effect of Cu addition near the solid solution limit at room temperature (Hansen, Anderko and Elliott 1958). To minimize the effect from other impurities, an extremely high purity Al (99.9996%) and OFHC Cu (99.99%) were used. However, it has been shown that for Al of such a high purity level that a fine scale structure cannot be obtained by plastic deformation as recrystallization takes place during or after cold rolling (Huang, Dong, Gong, Hansen, Liu and Huang 2012) or ARB processing (Kamikawa 2005). It is therefore one objective of the present study to investigate if the addition of a small amount of Cu solutes can prevent the occurrence of recrystallization and lead to the formation of a nanoscale structure after high strain deformation. Conventional cold rolling instead of ARB is applied to impose high strain deformation to the material in order to avoid any effect of ARB bonding interfaces. A second objective is to explore post deformation treatments in order to optimize strength and ductility.

2. MATERIALS AND METHODS

An Al-0.3wt%Cu alloy ingot was manufactured by melting and casting of Al and Cu. The ingot was then warm forged at 200°C to a slab of dimensions 300×300×50mm³. The slab was deformed by conventional cold rolling to 1 mm with a 98% reduction in thickness which corresponds to a von Misses strain of 4.5 ($\epsilon_{VM}=4.5$). In order to study the thermal stability, samples were annealed for 1h at temperatures in the range of 75 to 250°C. A sample was also annealed for 2h at 300°C to obtain a coarse grain size. The annealing treatments were done in a box furnace with a temperature control precision of $\pm 1^\circ\text{C}$. The annealed samples were cooled in water after annealing.

The microstructure in the as-cold-rolled state and after annealing treatments was characterized by means of transmission electron microscopy (TEM) and electron backscatter diffraction (EBSD). The longitudinal section (containing the normal direction, ND and the rolling direction RD of the processed samples was used for the microstructural characterization. For TEM observations, thin foil specimens were prepared by a twin-jet polishing method (Christiansen, Bowen and Lindbo 2002). The TEM observations were carried out with a JEOL 2000FX and a Zeiss Libra200 electron microscope both operating at 200 kV. For EBSD analyses, the samples were electropolished in a 10% perchloric acid and ethanol solution at -20°C and 20 V for 60s. The EBSD analyses were performed with a Zeiss Auriga dual beam station equipped with an Oxford Channel 5 EBSD System.

Mechanical properties were measured by tensile testing on a SHIMADZU Autograph AG-X 50kN electronic universal testing machine. Tensile specimens with a gauge dimension of 25mm long, 12mm wide, and 1mm thick were machined from the rolled and annealed sheets such that the tensile direction is parallel to the RD. Before tensile tests, samples were mechanically polished. Tensile tests were carried out at room temperature at an initial strain rate of $2 \times 10^{-4}/\text{s}$. Three to four specimens were tested for each condition to obtain average values of strength and

tensile elongation.

3. RESULTS AND DISCUSSION

3.1 Microstructure in the deformed state. Fig. 1a shows a TEM microstructure in the deformed state ($\varepsilon_{vM}=4.5$). A fine lamellar structure with the lamellar boundaries elongated along the RD was formed. The lamellar boundary spacings were measured along the ND from TEM images and the average spacing was about 200 nm, which is similar to the lamellar boundary spacing measured for commercial purity Al1100 alloys deformed by ARB to $\varepsilon_{vM}=4.8$ (Kamikawa 2005; Huang et al. 2006). Fig. 1b shows the EBSD map of the same sample. Similar to the TEM microstructure, the EBSD map also shows a lamellar structure although the details of the lamellar boundaries are not always well resolved. The distribution of boundary misorientation angles measured by EBSD is shown in Fig. 1c. It is seen that about 40% of the boundaries in the deformed state are high angles ($>15^\circ$). Note that the fraction of low to medium angle boundaries ($3-15^\circ$) is more than 50%, which is similar to what was observed in ARB processed high purity Al samples (Kamikawa, Tsuji, Huang and Hansen 2009). The boundaries having misorientation angles $<3^\circ$ are about 8%. The TEM and EBSD results demonstrate that a nanoscale lamellar structure has developed in the present Al-0.3%Cu alloy after cold rolling to a high strain ($\varepsilon_{vM}=4.5$). Clearly the occurrence of recrystallization during the cold rolling process has been completely suppressed which must be due to an effect of Cu solutes in stabilizing the microstructure (Gottstein 2005).

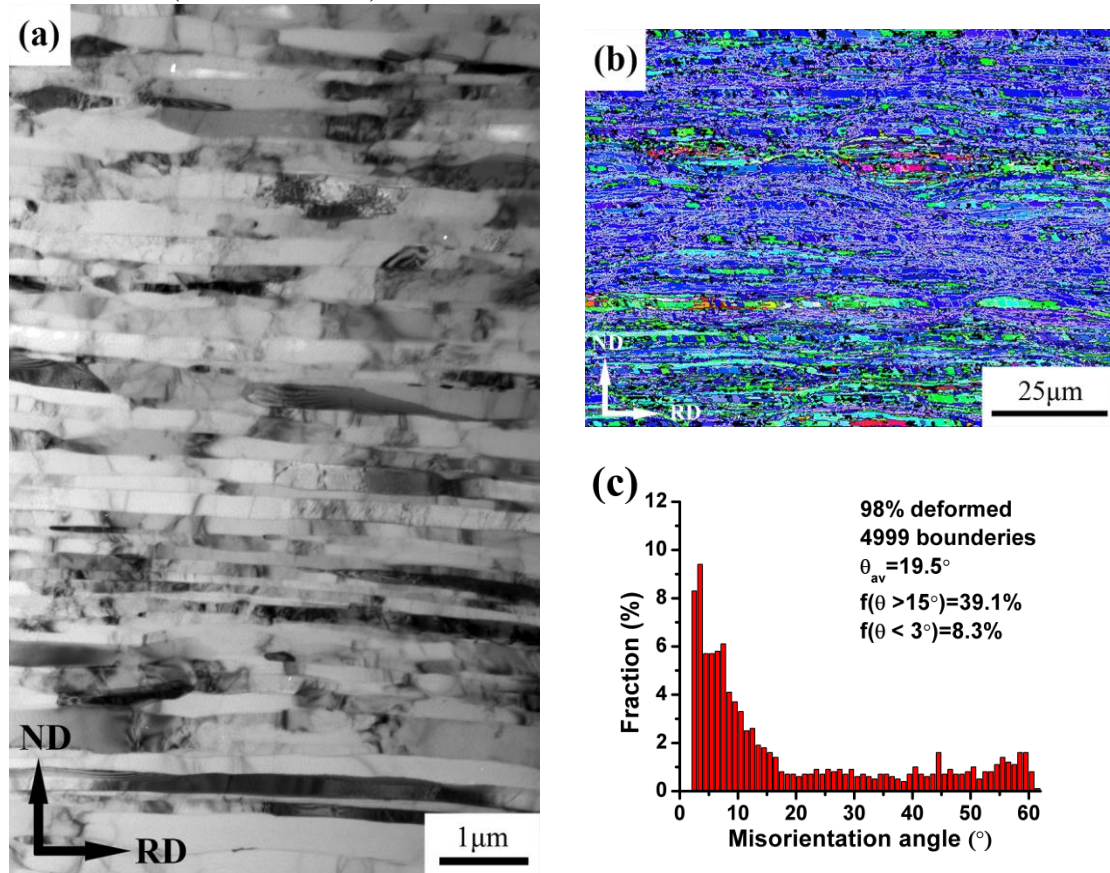


Fig.1. (a) TEM image, (b) EBSD image and (c) Distribution of boundary misorientation angles of 98% cold rolled Al-0.3%Cu alloy.

The lamellar boundary spacing of 200 nm measured for the present alloy with a nominal purity of 99.7% is similar to the lamellar boundary spacing measured for Al1100 alloy (99.2% pure)

deformed by ARB to $\varepsilon_M=4.8$ (Kamikawa 2005; Huang et al. 2006) but smaller than the value (about 400 nm) measured for Al1050 alloy (99.5%). This indicates that the addition of Cu has a stronger effect than other impurity elements present in the Al110 and Al1050 alloys in stabilizing the microstructure during cold rolling.

3.2 Structural evolution during annealing. The structural evolution during annealing at temperatures up to 300°C was analysed. It was found that after annealing at 75 to 150°C, the major change in the microstructure was structural coarsening and recovery without a significant change in the lamellar morphology. The microstructures of lightly annealed samples mainly consist of a lamellar structure mixed with a few equiaxed regions. After annealing for 1h at temperatures in a relatively narrow range 175 - 225°C, partial recrystallization occurred and the microstructure is mixture of some large grains (a few micrometers in size) and a fine deformed and recovered microstructure. The TEM and EBSD observations for the sample annealed at 175°C are shown in Fig.2a and 2b respectively, whereas the distribution of boundary misorientation angles is shown in Fig. 2c. After annealing at 250°C and higher temperatures, the sample is fully recrystallized.

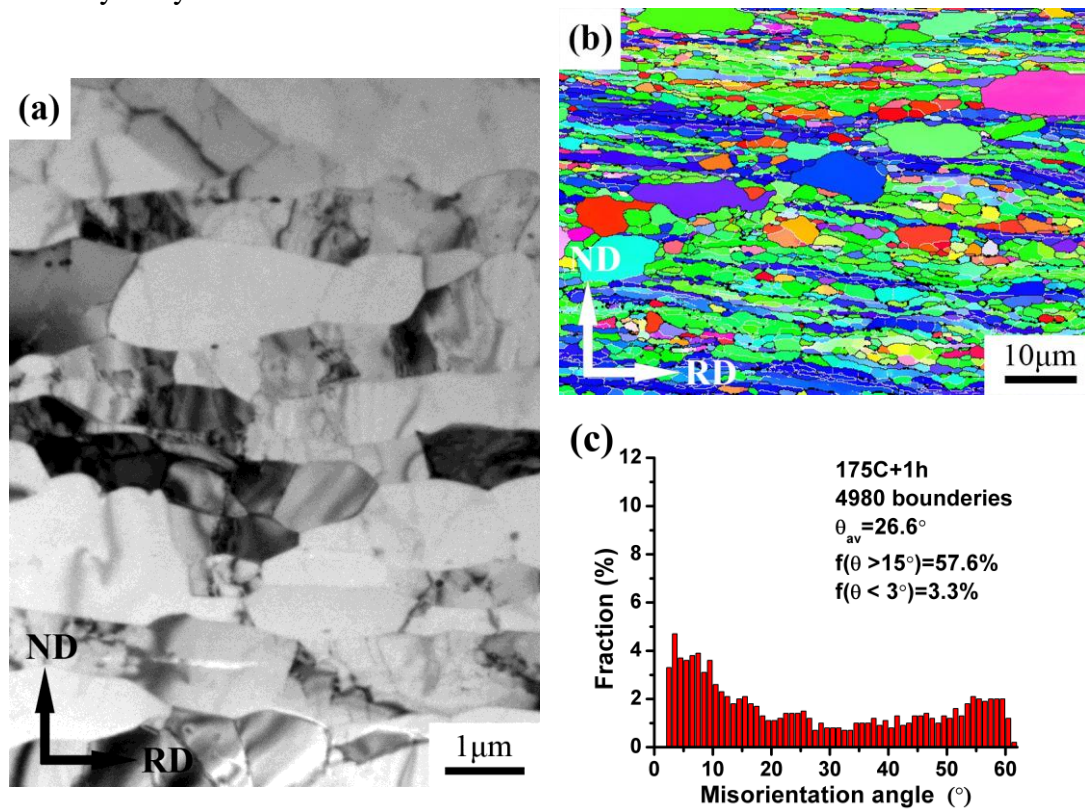


Fig.2. (a) TEM image, (b) EBSD image and (c) Distribution of boundary misorientation angle of annealed (175°C for 1h) Al-0.3%Cu alloy.

The evolution of boundary spacing during annealing was measured and the variation of average values as a function of annealing temperature is shown in Fig.3. The boundary spacing increases gradually when the annealing temperature increases up to 150°C. Note that the boundary spacings are still less than 1 μm at this stage. After partial recrystallization when annealing at 175-225°C, the samples have a bimodal microstructure. The sizes of recrystallized grains and recovered subgrains were separately measured and both sizes are shown in Fig. 3 (see the solid and open triangles). The size of recrystallized grains increases with a further increase in the annealing temperature and it reaches a value of about 60 μm after annealing for 2 h at 300°C.

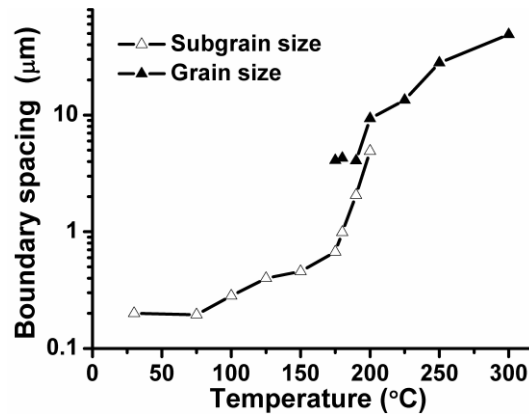


Fig.3. Boundary spacing/grain size versus annealing temperature of deformed Al-0.3%Cu.

3.3 Mechanical behaviour. Stress-strain curves of deformed and annealed samples are summarized in Fig.4. The deformed sample has a high strength (yield strength: 200 MPa; UTS: 225 MPa) and a total elongation of about 6%. The strength is in between the results reported for nanostructured Al1050 and Al1100 processed by ARB. The elongation is slightly lower than the reported value of about 7% for nanostructured Al1050 (Kidmose, Lu, Winther, Hansen and Huang 2012) and Al1100 (Kamikawa 2005; Huang et al. 2006). However, it should be pointed out that the gauge length of the tensile specimen (26mm) used in the present study is much longer than that (10 mm) used for the tensile testing of nanostructured Al1050 and Al1100 in the previous studies (Kamikawa 2005; Huang et al. 2006; Kidmose et al. 2012). As a larger total elongation is normally expected when the tensile gauge length is reduced (Zhao, Guo, Wei, Topping, Dangelewics, Zhu and Lavernia 2009), it is considered that the tensile ductility of the present nanostructured Al-0.3% Cu alloy is not worse than the nanostructured Al1050 and Al1100.

With an increase in the annealing temperature, the strength gradually decreases while the elongation shows little change in the annealing temperature range up to 150°C. This is in contrast to a sharp yield drop and a decrease in tensile elongation to <2% observed in the nanostructured Al1050 and Al1100 (Kamikawa 2005; Huang et al. 2006; Kidmose et al. 2012) annealed in the same temperature range. This indicates that the present nanostructured Al-0.3%Cu alloy shows an improved tensile stability as compared with the nanostructured commercial purity Al1050 and Al1100. After annealing at medium temperatures (e.g. 190°C), tensile stress-strain curves show a smooth work hardening behavior without the occurrence of yield point phenomena as observed in nanostructured Al1050 and Al1100. The total elongation is more than 15% and the yield strength still keeps at a level of about 100 MPa. This means that a good combination of strength and ductility can be achieved by annealing at medium temperatures.

Fig. 5 shows a Hall-Petch plot of the deformed and annealed samples. In the plot, the yield stresses were measured from tensile curves. As for the average boundary spacing/grain size, the values of deformed and 75-175°C annealed samples were determined from TEM micrographs, and those with annealing temperatures higher than 175°C were determined by EBSD analyses. The Hall-Petch slope was determined to be $81 \text{ MPa}/\mu\text{m}^{0.5}$, which is twice the Hall-Petch slope reported for pure Al in the coarse grained regime (Hansen 1977). This increased Hall-Petch slope may not necessarily mean an enhanced grain boundary strengthening. Instead, it is probably caused by a contribution of other strengthening mechanisms such as dislocation strengthening by low angle boundaries and the dislocations present in the volume between boundaries, as proposed by Hansen (Hansen 2004). The detailed strengthening mechanisms are

to be analysed in a forthcoming paper based on quantification of structural parameters including boundary spacing, misorientation angle and dislocation density.

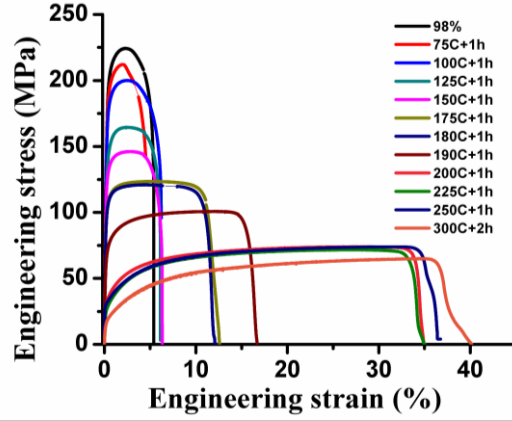


Fig.4. Stress–strain curves of Al-0.3%Cu samples deformed to $\epsilon_{vM}=4.5$ and then annealed at different temperatures.

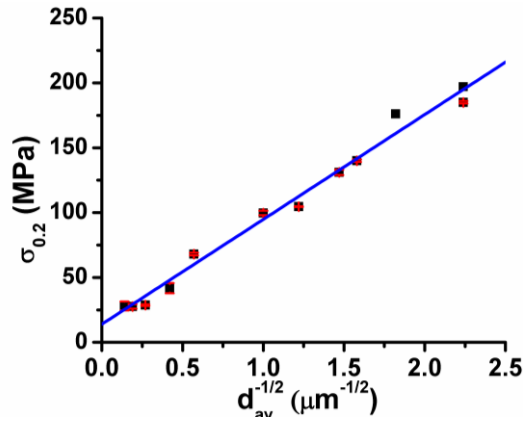


Fig.5. Hall–Petch plot of Al-0.3%Cu alloy processed by cold rolling and annealing.

4. CONCLUSIONS

1. The addition of 0.3wt% OFHC Cu solutes in high purity Al (99.9996%) effectively suppressed the recrystallization during the process of cold rolling, which led to the production of a stable lamellar structure with an average lamellar boundary spacing down to 200 nm and a high tensile strength of 225 MPa.
2. After low to medium temperature annealing, no enhanced tensile instability was observed as in the case of nanostructured commercial purity Al1050 and Al1100. A combination of relatively high strength and good ductility can be obtained i.e. a yield strength of 100MPa and a total elongation of 12 % after annealing at 175°C.

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